

UNITED STATES PATENT APPLICATION

OF

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AND

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FOR

**COMPOSITIONS COMPRISING AT LEAST ONE HYDROXIDE COMPOUND AND
AT LEAST ONE COMPLEXING AGENT, AND METHODS FOR USING THE
SAME**

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[0001] The present invention relates to compositions and methods for lanthionizing keratin fibers using a combination of at least one hydroxide compound and at least one complexing agent effective for dissociating the at least one hydroxide compound in sufficient quantity to effect lanthionization of the keratin fibers. The at least one complexing agent comprises at least one group chosen from groups of formula (I) as defined below and salts thereof.

[0002] Straightening or relaxing the curls of very curly hair may increase the manageability and the ease of styling such hair. In today's market, there is an increasing demand for hair care products referred to as "hair relaxers" which can relax or straighten naturally curly or kinky hair. Hair relaxers may either be applied in a hair salon by a professional or in the home by the individual consumer.

[0003] Hair fiber is a keratinous material which is comprised of proteins. Many of the polypeptides in hair fibers are bonded together by disulfide bonds (-S-S-). A disulfide bond may be formed from the reaction of the two sulfhydryl groups (-SH) one on each of two cysteine residues which results in the formation of a cystine residue. While there may be other types of bonds between the polypeptides in hair fibers, such as ionic bonds, the permanent curling and the shape of the hair is essentially dependent on the disulfide bonds of cystine residues.

[0004] Generally, hair relaxing processes are chemical processes which may alter the aforementioned disulfide bonds between polypeptides in hair fibers and may form lanthionine residues $[S[CH_2CH(NH-)(CO-)]_2]$. Thus, the term

"lanthionizing" is used when one skilled in the art refers to the relaxing of keratin fibers by hydroxide ions. "Lanthionizing," as used herein, refers to the formation of

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The result is the formation of lanthionine residues and HOS^- . See Zviak, C., The Science of Hair Care, 185-186 (1986). The second reaction sequence comprises at least one β -elimination reaction initiated by the nucleophilic attack of an available hydroxide ion on a hydrogen atom bonded to a carbon atom that is in the β -position with respect to the disulfide bond of a cystine residue. See Zviak. The result is the

These stable irreversible crosslinks in the treated hair make subsequent chemical re-linking of the polypeptides unnecessary. Thus, the only step that may be required following a straightening process using such hydroxide-containing alkaline agents is the removal of any excess alkaline solution to avoid or minimize damage to the hair protein or skin. If such a step is required, an acidic shampoo may be used to neutralize residual alkali and remove it from the hair and scalp.

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[0008] Other "no lye" relaxers may use hydroxide ions obtained, for example, from a slightly-soluble source, such as Ca(OH)_2 . For example, the slightly soluble Ca(OH)_2 may be mixed with guanidine carbonate to form guanidine hydroxide, a soluble but unstable source of hydroxide, and insoluble calcium carbonate (CaCO_3). This reaction is driven to completion by the precipitation of CaCO_3 and is, in effect, substituting one insoluble calcium salt for a slightly soluble calcium salt. Because guanidine hydroxide is unstable, the components are stored separately until the time of their use.

[0009] Guanidine carbonate and calcium hydroxide, however, create a different set of problems. The insoluble byproduct, CaCO_3 , leaves a white residue or unattractive "whitening" or "ashing" that remains in the hair since divalent metals like calcium have a relatively good affinity to keratin. A decalcifying shampoo is subsequently needed to remove the ashing.

[0010] Thus, there is still a need for a process to relax keratin fibers that has the advantages of using an insoluble or partially soluble metal hydroxide, such as $\text{Ca}(\text{OH})_2$, but reduces or eliminates the problem of ashing caused by the insoluble byproduct, CaCO_3 .

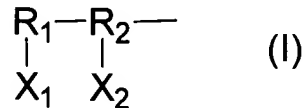
[0011] The present invention provides a method for lanthionizing keratin fibers comprising generating hydroxide ions in a composition, said step of generating comprising combining (i) at least one hydroxide composition comprising at least one hydroxide compound and (ii) at least one an activating composition comprising at

least one complexing agent effective for dissociating the at least one hydroxide compound in sufficient quantity to effect lanthionization of the keratin fibers. The composition comprising the generated hydroxide ions may then be applied to keratin fibers for a sufficient period of time to lanthionize the keratin fibers.

Lanthionization may be terminated when a desired level of relaxation of the keratin fibers has been reached. As used herein, "at least one" means one or more and thus includes individual components as well as mixtures/combinations. Further, "keratinous fibers" as defined herein may be human keratinous fibers, and may be chosen from, for example, hair.

[0012] According to the present invention the at least one complexing agent comprises at least one group chosen from groups of formula (I) and salts thereof.

Formula (I) is defined as



wherein:

- R₁ is chosen from a carbonyl group and a thiocarbonyl group;
- R₂ is chosen from CR groups wherein R is chosen from a direct bond to a neighboring atom, H, optionally substituted linear hydrocarbon groups, optionally substituted branched hydrocarbon groups, optionally substituted cyclic hydrocarbon groups, optionally substituted amino groups, optionally substituted thio groups, optionally substituted hydroxy groups, and halogen atoms;
- X₁ is chosen from hydroxyl groups and thiol groups; and

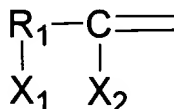
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- X₂ is chosen from hydroxyl groups, amino groups, and thiol groups.

For example, when R is a direct bond to a neighboring atom, groups of formula (I) have the following formula:



[0013] In another embodiment, the present invention is drawn to a composition comprising at least one hydroxide compound and at least one complexing agent effective for dissociating the at least one hydroxide compound in sufficient quantity to effect lanthionization of keratin fibers, wherein the at least one complexing agent comprises at least one group chosen from groups of formula (I) and salts thereof.

[0014] Another embodiment of the invention relates to a multicompartment kit comprising at least two compartments, wherein a first compartment contains a composition for generating hydroxide ions comprising at least one hydroxide compound; and a second compartment contains an activating composition comprising at least one complexing agent effective for dissociating the at least one hydroxide compound in sufficient quantity to effect lanthionization of keratin fibers.

The at least one complexing agent comprises at least one group chosen from groups of formula (I) and salts thereof.

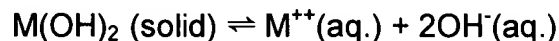
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[0015] It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory only and are not restrictive of the invention as claimed. Reference will now be made in detail to exemplary embodiments of the present invention.

[0016] Not to be limited as to theory, the lanthionization of keratin fibers is believed to be driven by the release of hydroxide ions, which disrupt the disulfide bonds of cystine. In one embodiment, the compositions of the present invention provide a novel way of generating soluble hydroxide ions from metal hydroxides wherein the soluble hydroxide ions are effective for relaxing hair. As described above, the hair relaxing compositions of the prior art utilized soluble metal hydroxides or slightly soluble metal hydroxides. Slightly soluble metal hydroxides, including most divalent metal hydroxides, are usually not soluble enough in water to generate sufficient soluble hydroxide ions to effect lanthionization of keratin fibers. This can be represented by the following, in which the equilibrium favors the left side of the reaction:



Therefore, in traditional relaxers containing slightly soluble metal hydroxides, the equilibrium was pushed to the right side and the reactions driven to completion by the precipitation of M^{++} as an insoluble compound, such as $CaCO_3$.

[0017] The compositions of the present invention, however, in one embodiment, utilize at least one complexing agent to dissociate the at least one hydroxide compound and to chelate and/or sequester M^{++} . The at least one complexing agent and the cation may form a complex that, in most cases, has a stronger interaction

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[0018] As described above, the at least one complexing agent according to the present invention comprises at least one group chosen from groups of formula (I) and salts thereof. Non-limiting examples of the R of R₂ include linear alkyl groups, branched alkyl groups, cyclic alkyl groups, linear alkylene groups, branched alkylene groups, cyclic alkylene groups, aryl groups, alkyl amino groups, alkylene amino groups, aryl amino groups, mercaptans groups, aryl thiol groups, hydroxyl groups, ether groups, ester groups, keto groups, and carboxyl groups. According to the present invention, R may also optionally be substituted.

[0020] Also note that as used herein, the term “alkyl group” refers to substituted linear alkyl groups, unsubstituted linear alkyl groups, substituted branched alkyl

[0021] Further, as used herein, “alkylene group” refers to substituted linear alkylene groups, unsubstituted linear alkylene groups, substituted branched alkylene groups, unsubstituted branched alkylene groups, substituted cyclic alkylene groups and unsubstituted cyclic alkylene groups, wherein the alkylene groups comprise at least one carbon and may optionally further comprise at least one heteroatom intercalated in the alkylene chain. Similarly, as used herein, the term “alkenyl group” refers to substituted linear alkenyl groups, unsubstituted linear alkenyl groups, substituted branched alkenyl groups, unsubstituted branched alkenyl groups, substituted cyclic alkenyl groups and unsubstituted cyclic alkenyl groups, wherein the alkenyl groups comprise at least one carbon and at least one double bond, and may optionally further comprise at least one heteroatom intercalated in the alkenyl chain.

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double bond, and may optionally further comprise at least one heteroatom intercalated in the alkenylene chain.

[0023] As used herein, "substituted" means comprising at least one substituent. Non-limiting examples of substituents include heteroatoms, such as oxygen atoms, sulfur atoms and nitrogen atoms, as well as functional groups, such as thiol groups, hydroxyl groups, ether groups, oxyalkylene groups, polyoxyalkylene groups, carboxylic acid groups, amine groups, amide groups, halogen containing groups, ester groups, siloxane groups, and polysiloxane groups. Thus, as used herein, substituted alkyl groups comprise, for example, hydroxylated alkyl groups (R-OH).

[0024] Non-limiting examples of the at least one complexing agent comprising at least one group of formula (I) include α -amino carboxylic acids (such as amino acids), α -hydroxy carboxylic acids (such as aldonic acids and aldaric acids), α -thio carboxylic acids, and α -hydroxy thioacids.

[0025] In one embodiment, the at least one complexing agent is chosen from mucic acid wherein mucic acid is optionally substituted; homologs of mucic acid wherein the homologs of mucic acid are optionally substituted; derivatives of mucic acid wherein the derivatives of mucic acid are optionally substituted; and salts of any of the foregoing. Mucic acid is a vegetable-derived polyhydroxy acid. Its chemical name is 1,2,3,4-tetrahydroxy-1,4-butane dicarboxylic acid, and it is manufactured, for example, by Soliance (Route de Bazancourt, F-51110 Pomacle, France) under the name MUCILIANCE. Mucic acid may be obtained by hydrolysis of the pectin contained in vegetable material to yield D-galacturonic acid. The electrochemical oxidation of D-galacturonic acid leads to mucic acid.

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[0029] The at least one complexing agent and the at least one hydroxide compound of the present invention may be present in a molar ratio ranging from 1 : 0.1 to 1 : 5 of the at least one complexing agent in the composition to the at least one hydroxide compound in the composition, where “the composition,” as used

herein, refers to the mixture of the at least one activating composition and the at least one hydroxide composition. In one embodiment, the at least one complexing agent and the at least one hydroxide compound of the present invention may be present in a molar ratio ranging from 1 : 0.3 to 1 : 2 of the at least one complexing agent in the composition to the at least one hydroxide compound in the composition. In another embodiment, the at least one complexing agent is present in an amount of 7% by weight relative to the total weight of the composition.

[0030] According to the present invention, the at least one hydroxide compound is chosen from alkali metal hydroxides, alkaline earth metal hydroxides, transition metal hydroxides, lanthanide metal hydroxides, actinide metal hydroxides, Group III hydroxides, Group IV hydroxides, Group V hydroxides, Group VI hydroxides, organic hydroxides, and compounds comprising at least one hydroxide substituent which is at least partially hydrolyzable. For example, the at least one hydroxide compound can also be chosen from those formed *in situ*, such as, for example, guanidine hydroxide. As previously mentioned, guanidine hydroxide may be formed *in situ*, for example, from the reaction of calcium hydroxide and guanidine carbonate. In one embodiment, the at least one hydroxide compound is chosen from calcium hydroxide, barium hydroxide, magnesium hydroxide, aluminum hydroxide, cupric hydroxide, strontium hydroxide, molybdenum hydroxide, manganese hydroxide, zinc hydroxide and cobalt hydroxide. In another embodiment, the at least one hydroxide compound is chosen from calcium hydroxide.

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[0031] According to the present invention, the at least one hydroxide compound may be present in an amount generally ranging from 0.1% to 10% by weight relative to the total weight of the at least one hydroxide composition, such as from 2% to 5% by weight.

[0032] In one embodiment, the at least one activating composition further comprises at least one compound effective for forming at least one salt of the groups of formula (I). A non-limiting example of the at least one compound effective for forming at least one salt of the groups of formula (I) is hydroxide compounds, which may be identical to or different from the at least one hydroxide compound. For example, the at least one compound effective for forming at least one salt of said groups of formula (I) may be chosen from sodium hydroxide, potassium hydroxide and lithium hydroxide. Further for example, the at least one compound effective for forming at least one salt of said groups of formula (I) may be chosen from organic bases. Non-limiting examples of organic bases include compounds comprising at least one quaternary ammonium group, compounds comprising at least one ammonium group, such as ammonium hydroxide, compounds comprising at least one amino group, such as amine bases, and guanidine and derivatives thereof. In one embodiment, the at least one compound effective for forming at least one salt of said groups of formula (I) is chosen from metal hydroxides, such as, for example, sodium hydroxide.

[0033] In another embodiment, the at least one activating composition further comprises at least one solvent. Non-limiting examples of the at least one solvent

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[0038] The compositions may also be provided in the form of a multicompartment kit. In one embodiment, the multicompartment kit comprises at

least two separate components. A first compartment of the kit comprises at least one composition for generating hydroxide ions that comprises at least one hydroxide compound. This first composition may, for example, be in the form of an emulsion, solution, suspension, gel or paste. A second compartment of the kit may comprise at least one activating composition comprising at least one complexing agent comprising at least one group chosen from groups of formula (I) and salts thereof, effective for dissociating the at least one hydroxide compound in sufficient quantity to effect lanthionization of keratin fibers. This second compartment may, for example, be in the form of an emulsion, solution, suspension, gel or paste. The skilled artisan, based on the stability of the composition and the application envisaged, will be able to determine how the composition and/or multicompartment compositions should be stored and mixed.

[0039] According to one embodiment of the invention, at least one compartment of the multicompartment kit will contains enough of at least one solvent to ensure that, upon mixing, enough of the generated hydroxide ions remain in solution to effect lanthionization of keratin fibers.

[0040] The at least one solvent useful in the present invention, in one embodiment, is a solvent that lowers the ionic bonding forces in the solute molecule enough to cause separation of the constituent ions. In one embodiment, the at least one solvent is chosen from water and organic solvents, such as dimethyl sulfoxide (DMSO).

[0041] The present invention also provides for a simple screening test, which may be used to determine the applicability of a complexing agent for use as the at

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least one complexing agent in the lanthionizing compositions of the present invention. By titrating a suspension of a hydroxide compound, such as $\text{Ca}(\text{OH})_2$, with the complexing agent of interest, the chelating or sequestering properties may be observed. If the solution reaches a pH sufficient for lanthionizing keratin fibers, then the complexing agent is a good candidate for use as the at least one complexing agent of the present invention.

[0042] The compositions of the present invention may further comprise at least one suitable additive chosen from additives commonly used in hair relaxing compositions. Non-limiting examples of the at least one suitable additive include dyes, anionic surfactants, cationic surfactants, nonionic surfactants, amphoteric surfactants, fragrances, screening agents, preserving agents, proteins, vitamins, silicones, polymers such as thickening polymers, plant oils, mineral oils, synthetic oils and any other additive conventionally used in compositions for the care and/or treatment of hair.

[0043] Other than in the operating example, or where otherwise indicated, all numbers expressing quantities of ingredients, reaction conditions, and so forth used in the specification and claims are to be understood as being modified in all instances by the term "about." Accordingly, unless indicated to the contrary, the numerical parameters set forth in the specification and claims are approximations that may vary depending upon the desired properties sought to be obtained by the present invention. At the very least, and not as an attempt to limit the application of the doctrine of equivalents to the scope of the claims, each numerical parameter

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should be construed in light of the number of significant digits and ordinary rounding approaches.

[0044] Notwithstanding that the numerical ranges and parameters setting forth the broad scope of the invention are approximations, the numerical values set forth in the specific examples are reported as precisely as possible. Any numerical value, however, inherently contains certain errors necessarily resulting from the standard deviation found in their respective testing measurements. The following examples are intended to illustrate the invention without limiting the scope as a result. The percentages are given on a weight basis.

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Example 1: Complexing of Solid $\text{Ca}(\text{OH})_2$ with Na_4EDTA

[0046] A solution of the complexing agent, 3 g of VERSENE 220 (tetrasodium EDTA (Na_4EDTA), 0.0066 moles) in 97 g of water, was titrated with the multivalent metal hydroxide solid, $\text{Ca}(\text{OH})_2$. At the end of the reaction, 0.60 g of $\text{Ca}(\text{OH})_2$ had been dissolved in the solution. Since the known solubility of $\text{Ca}(\text{OH})_2$ is 0.15g/100ml of water, the amount of $\text{Ca}(\text{OH})_2$ further dissolved in the solution due to the chelation of Na_4EDTA was 0.45 g or 0.0061 moles. The results are shown in Table 1.

[0047] Tetrasodium EDTA was a strong calcium binding constant in the high alkaline range. The results demonstrated that the complexing process occurs up to a 1:1 molar ratio of the complexing agent:metal hydroxide. The result was that the total chelation of $\text{Ca}(\text{OH})_2$ by Na_4EDTA at 1:1 molar ratio and the release of hydroxide ions to the solution. Since the solution reached a pH sufficient for lanthionizing keratin fibers, tetrasodium EDTA is shown to be an effective complexing agent.

Table 1: $\text{Ca}(\text{OH})_2$ Solubilized by EDTA

Amount of $\text{Ca}(\text{OH})_2$ added (gram)	pH	Appearance
0	11.62	Clear
0.20	13.32	Clear
0.40	13.52	Clear
0.60	13.59	Clear
0.65	13.63	Cloudy

Example 2: Generation of Hydroxide Ions from the Reaction of Mucic Acid and Calcium Hydroxide

[0048] To a solution of 7 g mucic acid (0.0333 moles) was added 2.66 g (0.066 moles) of sodium hydroxide. An incremental amount of calcium hydroxide was then added slowly to the slurry of mucic acid sodium salt, and the pH was measured. The results are shown in Table 2.

[0049] **Table 2: The Relationship between pH and Amount of $\text{Ca}(\text{OH})_2$**

Calcium Hydroxide Added	pH
0.0g	12.39
0.2g	13.02
0.4g	13.32
0.6g	13.46
0.8g	13.51
1.0g	13.53
1.2g	13.55

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[0050] The results indicate that hydroxide ions were generated when the insoluble calcium hydroxide was added to the mucic acid sodium salt slurry.

Example 3: Procedure for Measuring Relaxing Efficiency

[0051] A naturally kinky hair swatch was relaxed using a composition comprising at least one hydroxide composition comprising at least one hydroxide compound and at least one activating composition comprising at least one complexing agent according to the present invention. The relaxed hair swatch was then rinsed, shampooed, and then placed in the humidity chamber at 90% Relative Humidity (%RH) for 24 hours. The percent Relaxing Efficiency (%RE) is defined as:

$$\%RE = (L_f / L_t) \times 100$$

where L_f = length of the relaxed hair after 24 hours at 90% RH

L_t = length of the hair at the straight configuration

The following examples illustrate the method of using this new composition to straighten kinky hair.

Effectiveness of Mucic Acid in a Hair Relaxer

[0052] Mucic acid in 3 g of water was converted to its sodium salt by reacting with an appropriate amount of sodium hydroxide. This mucic acid sodium salt slurry was mixed with 6 g of a 7% calcium hydroxide cream. The calcium hydroxide cream contained the following ingredients by weight, relative to the total weight of the cream:

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[0054] The mixture of mucic acid sodium salt slurry and calcium hydroxide cream was applied to naturally kinky hair for 30 minutes. The treated hair was shampooed and then placed in a humidity chamber at 90% RH for 24 hours. The % RE is shown in Table 3.

Table 3: Relaxing Efficiency of Hair Treated with Mucic Acid and Calcium Hydroxide at Various Molar Ratios of Calcium Hydroxide:Mucic Acid

Ca(OH) ₂ Cream	Mucic Acid	50% NaOH	Water	Molar Ratio	%RE
6 g	1.19 g	0.9 g	3 g	1:1	21
6 g	0.84 g	0.64 g	3 g	1:0.7	86
6 g	0.60 g	0.46 g	3 g	1:0.5	80
6 g	0.48 g	0.37 g	3 g	1:0.4	81
6 g	0.36 g	0.27 g	3 g	1:0.3	54

The results indicate that mucic acid can be used with calcium hydroxide to relax naturally kinky hair.

Example 4: Effects of Water in Mucic Acid-Calcium Hydroxide Relaxer

[0055] Since the mucic acid sodium salt has limited solubility in water, additional water would increase the relaxing efficiency. Curly hair was treated with a slurry of mucic acid sodium salt with various amounts of water and 6 g of a 7% calcium hydroxide cream (see Example 3) (1:0.7 molar ratio of calcium hydroxide:mucic acid). The %RE are shown in Table 4.

Table 4: Relaxing Efficiency of Hair Relaxed with Mucic Acid and Calcium Hydroxide

Amount of Water in Mucic Acid Salt Slurry	%RE
2 g	42
3 g	88
4 g	95
5 g	98
6 g	99

[0056] The results indicate that as the availability of mucic acid salt increased, more hydroxide ions were generated, resulting in a higher relaxing efficiency.

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